

National Bureau of Standards



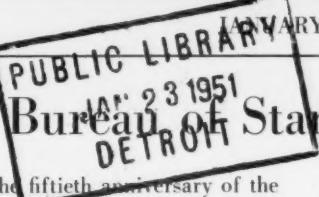
TECHNICAL NEWS BULLETIN

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National Bureau of Standards Semicentennial



The year 1951 marks the fiftieth anniversary of the establishment of the National Bureau of Standards. Created by act of Congress on March 3, 1901, the Bureau is the principal agency of the Federal Government for fundamental research in physics, mathematics, chemistry, and engineering. The Bureau's activities are thus largely concerned with the physical sciences although the close inter-relationships of the major fields of science preclude the drawing of sharp lines of distinction. Considerable work, for example, is undertaken in biochemistry and biophysics, particularly projects relating to materials, processes, and instrumentation.

The history of the Bureau actually extends back to the Constitution, which gave cognizance of weights and measures to the Federal Government, and the immediate antecedent of the Bureau was the Office of Weights and Measures, created by Congress in 1830. At the close of the last century, American industry and science recognized the need for a major, national physical laboratory similar to those established in England and Germany.

Problems relating to fundamental standards of science, physical constants, properties of materials, and methods and instruments of measurement called for a laboratory that would serve the entire nation. Industry, in particular, had needs for fundamental standards of measurement and ready access to a source of calibration of industrial and laboratory working standards. All working standards in research laboratories and industry are calibrated in terms of the national standards

of physical measurement that the Bureau maintains. In addition, the Bureau carries on necessary research leading to improvements in such standards and in related measurement methods and instruments. New standards, methods, and instruments, in keeping with demands for greater precision and the opening up of new fields of science and industry, are also objects of research.

An equally significant aspect of the Bureau's work is that concerned with specific research and development projects in the physical sciences and mathematics. Some of these projects are related to fundamental standards, properties of materials, and physical constants, as well as measurement problems. For example, the development of the atomic clock is directly related to the Bureau's interest in a better standard of time and frequency, and it is also linked to problems in the field of microwave spectroscopy. Others of these projects range in subject matter from pure research in mathematics, in nuclear physics, and in supersonic aerodynamics to the development of guided missiles, proximity fuzes, and automatic electronic computing machines.

The scope of the Bureau's work is suggested by the names of its 15 divisions: electricity, optics and metrology, heat and power, atomic and radiation physics, chemistry, mechanics, organic and fibrous materials, metallurgy, mineral products, building technology, applied mathematics, electronics, ordnance development, radio propagation, and missile development. The divisions in turn consist of several sections—totaling 108—organized as logical units of the divisions. In ad-

dition to laboratory work, the Bureau, by virtue of its staff and facilities, is called upon to render scientific advisory services to the Government and industry. Its participation in the activities of scientific and engineering societies and bodies, both nationally and internationally, is necessarily extensive and varied.

A large number of the principal scientific and technical organizations of the nation, in recognition of the role of the Bureau in science, have planned meetings in Washington in 1951 to honor the Bureau's Semicentennial. The present calendar of major meetings is presented below. Some of the meetings have regularly been held in Washington in the past; most of them were planned for Washington by the societies in view of the Bureau's Semicentennial.

Principal Organizations

- American Society of Photogrammetry, January 10 to 12.
Joint AIEE-IRE-NBS Conference on High Frequency Measurements, January 10 to 12.
American Crystallographic Association, February 15 to 17.
Inter-Society Color Council, February 28.
Optical Society of America, February 28 March 1 to 3.
Electrochemical Society, April 8 to 12.
Union Radio Scientifique Internationale, April 16 to 18.
National Academy of Science, April 23 to 25.
American Physical Society, April 26 to 28.
APS Division of High Polymer Physics, April 26 to 28.
Bone Char Conference II, May 3 to 4.
Acoustical Society of America, May 10 to 12.
Horological Institute of America, May 14 to 15.
Society for Experimental Stress Analysis, May 16 to 18.
National Conference on Weights and Measures, May 22 to 25.
Analytical Division of the American Chemical Society, June 14 to 16.
American Astronomical Society, June 21 to 23.
Illuminating Engineering Society, August 26 to 31, September 1.
International Union of Chemistry, September 13 to 15.
American Roentgen Ray Society, September 25 to 28.
American Dental Association, October 15 to 20.
National Institute of Governmental Purchasing, October 21 to 24.
Institute of Mathematical Statistics, October 26 to 27.
American Mathematical Society, October 27.
Conference on Electrical Insulation (NRC), October 29 to 31.
APS Division of Electron Physics, November 1 to 3.

In addition, a number of committee conferences and meetings of local sections of societies will be held at the Bureau:



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U. S. DEPARTMENT OF COMMERCE

CHARLES SAWYER, *Secretary*

NATIONAL BUREAU OF STANDARDS
E. U. Condon, *Director*

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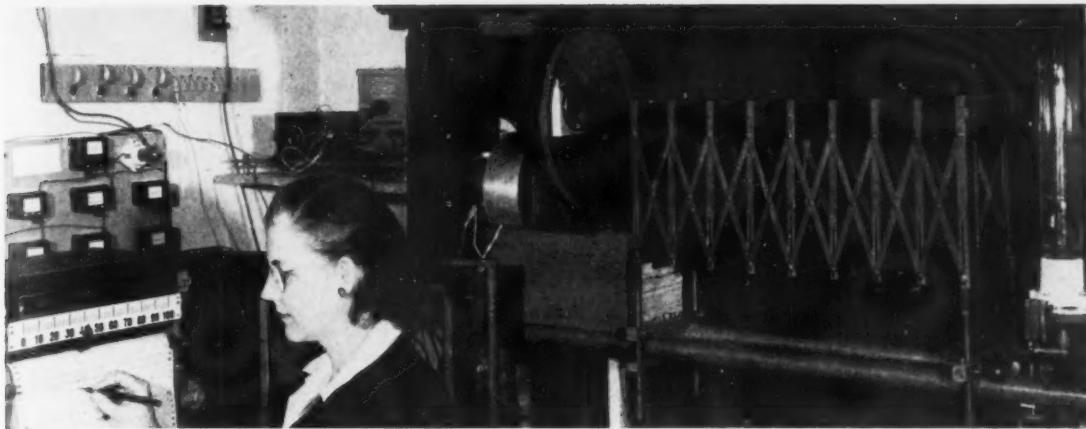
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Committees and Local Sections

- Electrochemical Society (Baltimore-Washington Section), January 18.
ASTM Committee D-1 on Paint, February 26 to 28.
ASTM Committee E-12 on Appearance of Engineering Materials, February 27.
Electrochemical Society (Baltimore-Washington Section), March 15.
ASTM Committee D-14 on Adhesives, April.
American Ceramic Society (Baltimore-Washington Section), April 2.
ASTM Committee B-1 on Electrical Conductors, April 19 to 20.
Electrochemical Society (Baltimore-Washington Section), May 17.
ASTM Committee E-3 on Chemical Analysis of Metal, June 13 to 14.
Mathematical Association (Maryland-Washington-Virginia Section), December 8.
The Bureau is also planning a number of symposia on special topics to be held during the year.



Improvements in the NBS physical photometer have made it possible to reevaluate the candlepower and luminous flux of mercury-vapor lamp standards (extreme right) with greatly increased certainty. The principal elements of the photometer are the luminosity filter and the thermopile, which is mounted in a large brass black body. A rotating disk gives equal periods of "light-on" and "light-off". These are grouped together in the center of the photograph.

New Flux Values for Mercury Vapor Lamps

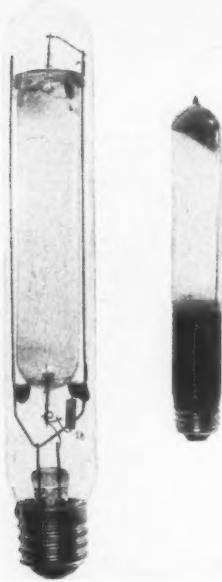
The National Bureau of Standards announces new luminous flux values for mercury-vapor lamps derived from the new photometric units that were formally established in this country by law (1) in July 1950. Based on the work of L. E. Barbow and associates of the Bureau's photometry and colorimetry laboratory, the revaluation places mercury lamps on a basis consistent with incandescent and fluorescent lamps. Standards of candlepower or luminous flux for the incandescent and fluorescent lamps issued or calibrated by the National Bureau of Standards have been based on the new units since 1948, when such values were first introduced in the United States following international agreement (2). For incandescent lamps the changes in values thus brought about were relatively unimportant, being less than 1 percent for lamps in common use. No changes were necessary for fluorescent lamps because their development has been so recent that they have always been calibrated in terms of the new units, originally proposed in 1938. Changes in the rated values for mercury lamps, however, are as large as 9 percent for one type.

The chief advantages of the new system of photometric units are establishment of the units on a reproducible physical basis and the international standardization that is effected at the same time. In this system the unit of intensity of light is the candle, which is one-sixtieth of the intensity of 1 square centimeter of a perfect radiator (known as a "black body") when operated at the temperature of freezing platinum. The unit of flux of light is the lumen or the flux in a unit of solid angle from a source having an intensity of one candle. The candlepower and luminous flux of lights whose colors are different from the primary standard are evaluated by means of standard luminosity factors

representing the special sensitivity of the average eye under specified conditions.

The mercury-lamp standards involved in this revaluation are the 400-watt and the 250-watt sizes. They were originally evaluated in 1936 by direct visual comparison with the incandescent-lamp standards in force at that time. Such comparison, however, poses one of the most difficult of visual photometric problems because the spectral distributions of radiant flux in the two types of light sources are of such radically different types. The incandescent light gives a continuous spectrum throughout the whole visible range. Light from the mercury arc, on the other hand, is concentrated largely in a few lines in the yellow, green, blue, and violet regions of the spectrum. Even though calibrated filters are placed over the incandescent lamps to give an approximate color match, there still remain the usual individual observer differences common to heterochromatic photometry but accentuated in this case by possible large differences in macular pigmentation among the observers. Although eight observers were used in the 1936 measurements and the photometric conditions such as field size and luminance (photometric brightness) were those accepted as standard for such work, the final values were unavoidably subject to considerable uncertainty, as recognized at that time.

The development of an accurate physical photometer (3) by the National Bureau of Standards has made it possible to reevaluate the candlepower and luminous flux of these mercury vapor lamp standards with greatly increased certainty. Although the idea of the physical photometer is not new, its development to the present state of perfection by the Bureau is largely responsible for the improved accuracy. There are two principal elements in this instrument: the thermopile



Mercury-vapor lamps of the type maintained as photometric standards at the National Bureau of Standards: (Left) 400 watts, (right) 250 watts.

detector with its amplifying and recording attachments and the luminosity filter by means of which the radiant flux incident on the detector is weighted to give a response proportional to that of the average human eye.

Major improvements have been introduced into both of these elements since the equipment was first designed in 1941. The sensitive galvanometer and potentiometer originally used have been replaced by a photoelectric self-balancing potentiometer and amplifier; the output is used to operate a recorder. The use of a sectored disk driven by a synchronous motor gives equal periods of "light-off" and "light-on". The recorder automatically provides a record of the readings and plots a graph to determine the response, without the necessity of balancing the potentiometers.

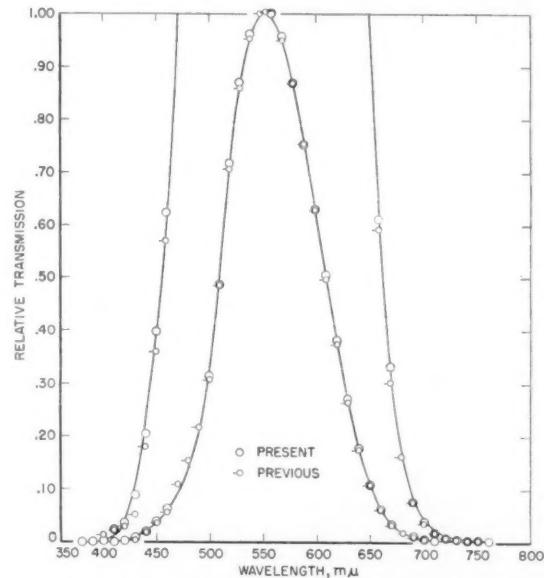
Since the blackened thermopile is nonselective in its spectral response, the filter must closely duplicate the standard ICI luminosity factors (4) if photometric results obtained on the physical photometer are to conform with the new photometric units. The most recent design of this filter (5) at the Bureau completely eliminates stray radiant flux from the infrared while maintaining excellent duplication of the ICI luminosity factors. With the new filter, there is no need to apply the somewhat uncertain correction for stray infrared flux.

Direct determination of the luminous flux of a lamp by means of a white-lined integrating sphere is not possible with high accuracy unless a carefully calibrated lamp of the same type and approximate size is available to serve as a standard by the substitution procedure. For an initial standard, therefore, it is necessary to measure, first, the intensity (candlepower) in a

specified direction—in this case with the physical photometer as noted above—and, second, the distribution of intensity in all directions from the lamp. The lumen output may then be computed from these measurements by a summation procedure. The lumen value assigned to the lamp will thus be based on the same units and be subject to the same uncertainties as apply to the intensity values.

In the 1936 evaluation the ratio of the luminous flux of each lamp to its candlepower in a direction perpendicular to the plane of the supports within the lamp was determined. For the six 400-watt lamps these ratios averaged approximately 10.4, ranging between about 10.2 and 10.5. For the six 250-watt lamps the corresponding ratio averaged approximately 10.2, ranging from about 10.0 to about 10.3.

Distribution measurements of lamps necessitate operating the lamps for relatively long periods, and it was decided that new distribution measurements would not be made on the original group of mercury-vapor standards, but that an indirect method would be used to check the previously determined ratios of luminous flux to candles in the specified direction. A new group of six 400-watt mercury-vapor lamps was measured on a distribution photometer equipped with a photoelectric cell, in which the distance between the cell and lamp is approximately 25 feet. The luminous intensity of each of these lamps was determined with the new NBS physical photometer. An intercomparison of the old and new standards in the NBS photoelectric integrating



Relative spectral transmittances of two luminosity filters (plotted points) are compared with the standard ICI luminosity factors (continuous curve). A slight transmittance of infrared radiant flux in the former filter is completely eliminated in the present version. The outer curve is a magnification ($\times 10$) of the ends of the ICI curve.

sphere indicated excellent agreement between the luminous flux values of the two sets of lamps assigned on the basis of (1) the new distribution photometer and new physical photometer measurements of the new lamps, as against (2) the old distribution photometer and new physical photometer measurements of the original group of lamps.

The new values assigned to the 250-watt mercury-vapor lamps are 4.5 percent lower than those formerly assigned visually, whereas the new values assigned to the 400-watt mercury-vapor lamps are 9 percent lower. An intercomparison of these two groups of standards in the integrating photometer showed excellent agreement. This verifies the estimate that the uncertainty in the new values assigned to the mercury-vapor lamp standards does not exceed 2 percent, divided about 1 percent for the physical photometer measurements of candle-power and an additional 1 percent for the conversion from candles to lumens. Manufacturers and others interested in mercury-vapor lamps have been kept in-

formed of the progress of this work by correspondence and conferences. Results obtained at another laboratory compare favorably with those obtained at this Bureau. By mutual agreement, effective August 1, 1950, all calibrations of mercury-vapor lamps are on the new basis.

- (1) Public Law 617, 81st Congress (July 21, 1950); Also NBS Technical News Bulletin **34**, 135 (Sept. 1950).
- (2) Announcement of changes in electrical and photometric units, NBS Circular C459 (1947). Effect of changes in electrical and photometric units or previously assigned values for standard lamps, NBS Letter Circular LC364 (1947).
- (3) R. P. Teele, A Physical photometer, J. Research NBS **27**, 217 (1941) RP1415.
- (4) These factors were adopted in 1924 by the International Commission on Illumination as a result of work at the NBS. A résumé of that work and a discussion of subsequent developments are given in a paper by K. S. Gibson, J. Opt. Soc. Am. **30**, 51 (1940).
- (5) R. P. Teele and K. S. Gibson. A standard luminosity filter, J. Opt. Soc. Am. **38**, 1096 (1948).

Absorption of Oxygen by Alkaline Pyrogallol

One of the most important reactions in gas analysis, believed for many years to be subject to unavoidable errors, has been found dependable by chemists of the National Bureau of Standards when a few simple conditions are met. This is the determination of oxygen by absorption in an alkaline pyrogallol solution. It has long been recognized that carbon monoxide is formed during the reaction, and the analysis therefore shows less oxygen than is actually present. However, it has not been possible to correct for the carbon

monoxide because the quantities produced are variable and unpredictable. Investigation of the problem by Marthada V. Kilday of the Bureau's gas chemistry laboratory has resulted in a procedure that improves the accuracy of the analysis.

The first step in the investigation was the selection of a suitable method for measuring the amount of carbon monoxide formed in any one reaction. Only when this was known could the conditions causing its production be found and corrected.

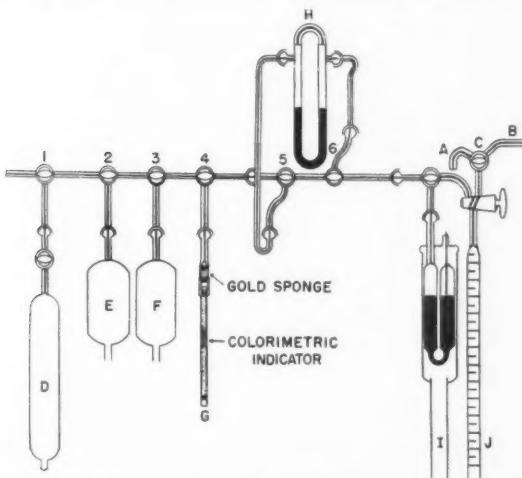
For this purpose, the NBS Carbon Monoxide Indicator (NBS Technical News Bulletin No. 354, 73 (1946)), developed during World War II, proved particularly suitable. The indicator, a yellow chemical that turns green on exposure to carbon monoxide, can be compared with known standards to give quantitative readings. It is most accurate for concentrations of carbon monoxide up to 0.1 percent. Higher concentrations can be determined by diluting the sample.

A modification of the Shepherd gas analysis apparatus was used for the test reactions. Oxygen in known mixtures was absorbed in pyrogallol, and the residue was passed through the indicator. Here the amount of carbon monoxide was determined so that corrections could be made for it in the final computations.

The greatest source of error was found to be the method of preparing the pyrogallol solution. Heat produced by the reaction, if not carried away immediately, resulted in the formation of carbon monoxide in unpredictable amounts.

As a result of this work, the accuracy, reproducibility, and rapidity of the determination of oxygen by absorption in pyrogallol may be improved under the following conditions:

1. Solutions can be prepared that evolve no significant amount of carbon monoxide during the usual



Conventional gas analysis apparatus was modified to study the conditions affecting the formation of carbon monoxide during the absorption of oxygen by alkaline pyrogallol solutions. The modifications include reservoirs E and F for mixing and diluting samples high in carbon monoxide, carbon monoxide indicator tube G (with gold sponge to remove mercury vapor), and flowmeter H.

analysis. This is accomplished by the direct addition of a saturated solution of potassium hydroxide to pyrogallol crystals while the reaction vessel is immersed in ice water and closed to the atmosphere.

2. Samples having partial pressures of oxygen higher than 50 percent can be analyzed with the *improved* solutions without dilution of the sample.

3. A pipette that disperses the gas in fine bubbles through at least 18 centimeters of solution is preferable.

4. The rate of flow of the sample into the pyrogallol

should be as rapid as possible and never less than 20 milliliters per minute.

5. The temperature of the pyrogallol reagent should not exceed 30° C. during absorption.

6. If high accuracy is desired, the solution should not be used for analysis after 12 volumes of oxygen have been absorbed per volume of solution.

For further technical details, see A quantitative study of the carbon monoxide formed during the absorption of oxygen by alkaline pyrogallol, by Marthada V. Kilday, J. Research NBS 45, 43 (1950) RP2112.

Sampling of Upper Leather for Shoes

Scientists of the National Bureau of Standards have developed basic information on shoe upper leather that promises to eliminate laborious and wasteful testing of individual hides. The results make it possible for manufacturers and purchasers of leather for shoes to draw up a variety of sampling procedures designed to suit any predetermined set of conditions under which the leather may be selected. This project was inaugurated by the Bureau at the request of the Office of the Quartermaster General as part of a program aimed at supplying better footwear for the Army; it was undertaken by Edwin B. Randall and his associates of the Bureau's Leather Section and John Mandel, a Bureau analytical statistician, with the cooperation of Charles W. Mann of the Office of the Quartermaster General.

The purpose of the Bureau's study was to determine the number of hides that must be taken from a "lot" of leather in order to obtain a representative sample, and to discover how many specimens should be taken for test from each side of leather.

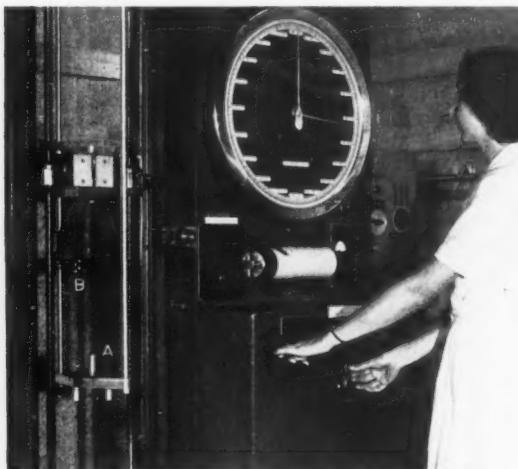
Material for the investigation consisted of 30 sides

of chrome-tanned leather, of medium chrome and grease contents, split to 5½-ounce weight. From each side, 21 blocks were cut so that corresponding locations on all the selected sides might be compared. The physical and chemical tests chosen for the study were those generally applicable to shoe upper leather—i. e., tensile strength, elongation, stitch tearing strength, tongue tearing strength, bursting strength, density, water penetration, water-vapor permeability, chromic oxide, hide substance, and grease. For full strength tests, in which a directional effect was expected, specimens were cut in two directions, parallel and perpendicular to the backbone.

One of the first important results was the discovery that physical and chemical properties vary more widely between different locations on the same side than between corresponding locations on different sides. Though this fact had previously been established for heavy leather by workers at the Bureau, the present work is the first demonstration of its applicability to upper leather.

Since, in production, all of the 21 locations of each side cannot be tested, as they were in the Bureau's investigation, the importance of determining the best testing locations cannot be overemphasized. It is clear that a location should be selected whose properties reflect most adequately those of the entire side. This criterion can be understood in various ways. For example, one may search for a location whose properties come closest to the average for the side. Or, on the other hand, a location may be found whose properties, even though they are quite different from the average for the side, bear some constant relation to the latter. On the basis of theoretical consideration and of a careful study of the data, it was concluded that the most desirable sampling location is that for which the properties bear a constant relation to those of the side.

The investigation proved that this criterion of constancy could not be met simultaneously for all properties by the selection of a single location. For each property, however, there are locations for which that property is, except for relatively small fluctuations, a linear function of the average of the side. It was shown that for any property the definiteness of this linear relationship can be measured by a well-known statistical function: the coefficient of correlation between the value



In order to test the burst strength of leather samples too tough for ordinary testing machines, an attachment was devised (left) for a Tinius Olsen Testing Machine. The force required for the plunger (A) to burst the sample (B) is recorded on the revolving drum (upper center).

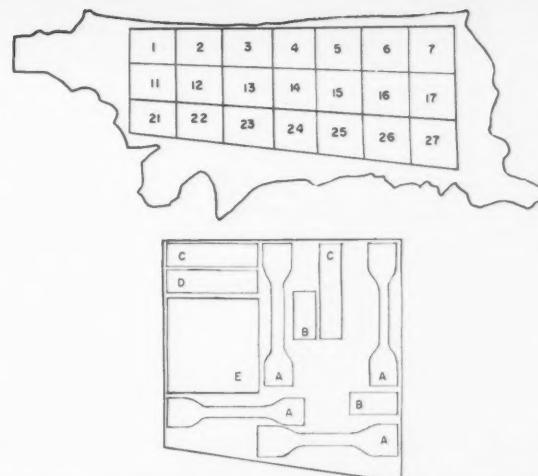
for the selected location and the corresponding average value for the entire side.

On the basis of these considerations, a formula was derived giving a mathematical relationship between the coefficient of correlation of the selected location, the confidence with which the difference in properties between two lots is to be judged, and the number of sides required for that purpose. For each property and for each possible sampling location the correlation coefficient is calculated between the test result for that location and the average of all test results (for the property considered) for the entire side. The position for which this coefficient is largest is the most suitable sampling location for the property considered.

As a result of this study, a sampling procedure was devised for upper leather currently under experimental study by the Bureau for the Office of the Quartermaster General. In the selection of the sampling location for a particular test, consideration was first given to the locations from which the fewest specimens would be required to give the desired level of accuracy. However, other factors were considered, such as the desirability of grouping the test specimens in a small area in order to minimize the destruction of leather in other areas designated for storage and wear tests.

Use of only one specimen per side from the proper sampling location, instead of one specimen from each of the 21 blocks, may result in savings of as much as 95 percent in leather and labor without sacrificing accuracy. The higher the accuracy required, the larger will be the number of sides that must be sampled in order to detect differences between lots.

The test methods used in the work are described in Federal Specification KK-L-311 with two important exceptions. One is the water vapor permeability test



A side of chrome-tanned leather was divided into 21 blocks (above) from which specimens were cut (below). These specimens were subjected to tests of tensile strength and stretch (A); stich tear (B); tongue tear (C); density (D); and bursting strength, water-vapor permeability, and resistance to water penetration (E).

devised by Kanagy and Vickers of the National Bureau of Standards. The other is a special adapter developed at the Bureau for use with the Tinius Olsen Testing Machine to measure bursting strength. This adapter was used instead of the Mullen tester because the latter did not have enough capacity to break all of the side leather specimens in this experiment.

Note: Water-vapor permeability of leather, NBS Technical News Bulletin 34, 163 (1950).

Labeling Sugar With Radioactive Carbon

The National Bureau of Standards has begun a new project designed to prepare sugars and other carbohydrates with radioactive carbon atoms located in specific positions within the molecule. Such accurately labeled sugars will be a powerful tool for biological and medical researchers, enabling them to trace a single carbon atom through an entire series of complex chemical reactions.

Under the direction of Dr. H. S. Isbell of the Organic Chemistry Section, the Bureau will develop synthesizing methods and will prepare samples of sugars tagged with carbon 14 atoms. These sugars in turn can be used to make labeled samples of other carbohydrates, such as starch or cellulose. The program, sponsored by the Atomic Energy Commission, has been initiated to meet the increasing need for carbohydrates that can be traced through the processes of living cells. Labeled sugars prepared at the Bureau will have particular application in an investigation of the synthesis of labeled cellulose initiated by Dr. Glenn A. Greathouse of the National Research Council.

Previous studies of carbohydrate metabolism by

tracer techniques have been largely restricted for lack of a source of labeled sugars. Introducing the C¹⁴ atoms at specific points within the sugar molecule has until now proved to be a highly intricate and inefficient process with yields of less than 10 percent recoverable radioactive material. The NBS group hopes to improve the methods of production sufficiently to make the sugars containing specifically located C¹⁴ available to other laboratories.

At first, the Bureau scientists will concentrate on preparing glucose, fructose, and lactose, the more common sugars, with C¹⁴ atoms in the carbon 1 position. Later, however, the program will be extended to include other sugars and other positions.

The specific activity of labeled sugars will be so great that their radioactivity will be detectable in a dilution of one part in a million. The ease with which the tagged atoms can be traced and the restriction of C¹⁴ to specific points in the molecular structure gives the biological researcher an extremely powerful means of studying the many complex reactions and interconversions that occur in living organisms.

The requirements of modern commerce and industry make uniformity in standards of mass throughout the Nation no less important than uniformity of currency. Obviously, an even higher degree of uniformity is necessary among reference standards of mass kept by research laboratories to maintain high accuracy in scientific weighing.

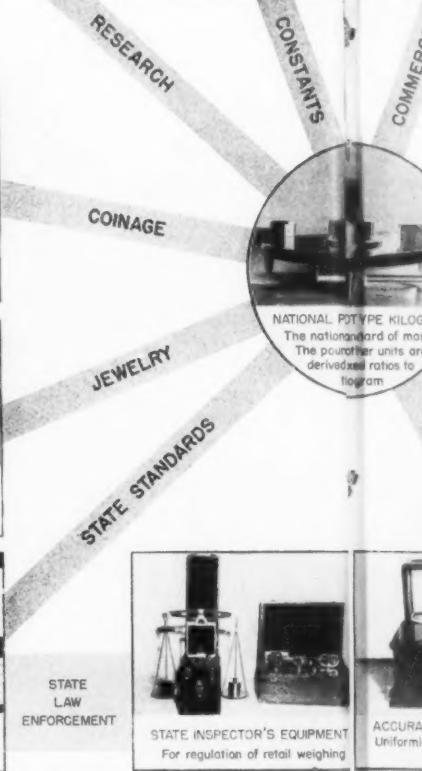
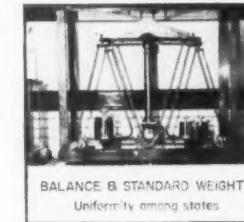
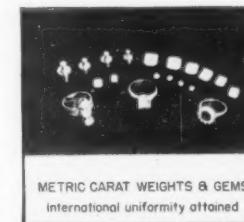
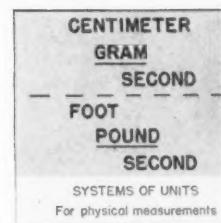
High-Precision Measurements

The national standard of mass—a platinum-iridium cylinder about 1½ inches high, 1½ inches in diameter, and having a mass of almost exactly 1 kilogram—is maintained in a special vault of the National Bureau of Standards. The United States standard kilogram, like the standard meter bar, is a copy of an international standard kept at the International Bureau of Weights and Measures at Sevres, near Paris. It is known as Prototype Kilogram No. 20. This prototype was established as the national standard by executive order in 1893. Since that time, the pound and other everyday units of mass have been defined by the ratios of their mass to the mass of this kilogram. Occasionally the national standard kilogram is removed from the vault to check the best secondary standards. These include other platinum-iridium standards from 1 kilogram to 0.05 milligram and two 1-kilogram standards of nickel-chromium alloy. From the secondary standards, values are derived for the working standards in the various customary and metric units used in everyday testing in the Bureau's laboratories.

Weights submitted to the Bureau for calibration include the commercial classes of standards primarily used as the basis of State regulation of weights and measures, the laboratory classes of standards used in scientific research and development, and weights of special design required for industrial or technical work. In some classes of weights such as those for precise balances, actual values are determined to the required precision; in others, such as weights for testing master track scales, it is only necessary to verify accuracy within specific tolerances. The Bureau's mass laboratory also investigates the degree of constancy and sources of variability in weights and makes studies to designate materials having good corrosion resistance, nonmagnetic properties, suitable density, and other desirable properties for weights. It is thus able to specify types of construction and tolerances for the different classes of weights.

The most accurate of the precision balances used by the Bureau in its calibration work can compare two platinum or platinum-iridium kilogram weights with an error of less than one part in 100,000,000. To make this high precision possible, the balance is kept in an air-conditioned room beneath the surface of the ground, and errors due to the heat of the human body are prevented by operating the balance entirely by remote control. Not only is the balance beam arrested and released in this way, but the interchanging of the loads on the pans and the addition of sensitivity weights are also accomplished without human intervention.

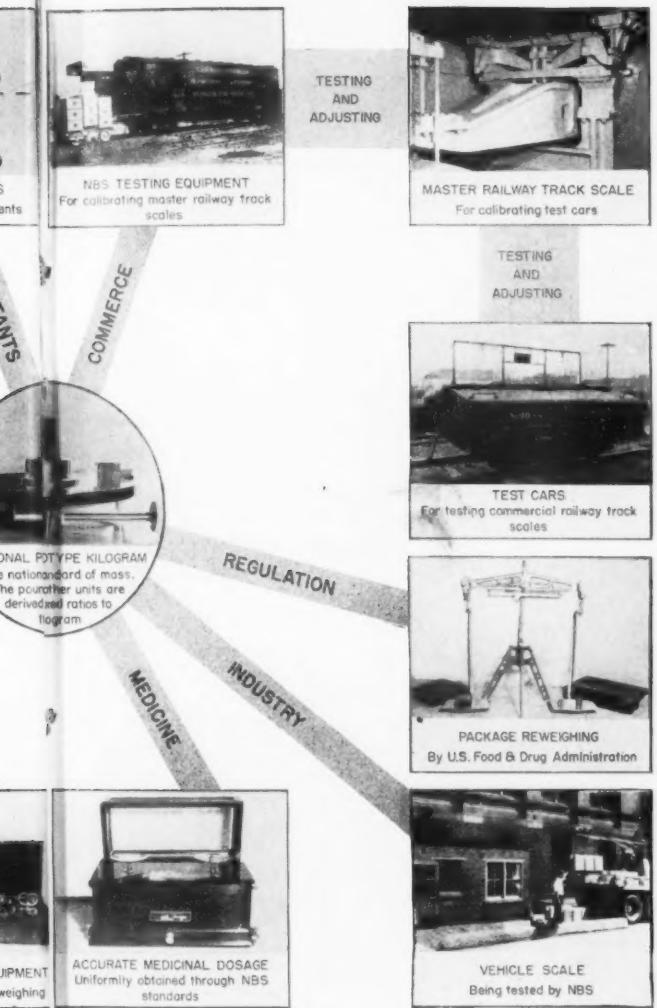
Standards of



The national standard of mass, of which the Bureau is the custodian, provides the basis for the regulation of weights and measures throughout the United States.

In determining the exact values of the best secondary standards of mass, the Bureau has found many procedures necessary that are not ordinarily employed in weighing. Thus, elaborate care is required in the determination of air-buoyancy effects when the best secondary kilogram standards are compared with the standard platinum-iridium kilogram. The air-buoyancy effects are important because of the difference in material of the weights being compared. Most of the standard weights in use in scientific research, as well as in commercial regulatory work are of brass or some alloy of similar density, and the difference in air buoyancy for brass and platinum or platinum-iridium kilo-

Standards of Mass



The standard provides a basis for uniform weight in science, commerce, and industry.

gram standards amounts to about 75 mg. Two finely finished nickel-chromium kilogram standards, calibrated by comparison with the prototype kilogram in such a way as to include the necessary correction for air buoyancy in their apparent mass, are used in calibrating brass weights. In the calibration of these nickel-chromium standards, the density of the air is determined with great care. Barometric pressure is accurately measured by means of a sensitive and highly reliable aneroid barometer especially designed for this work. Readings of the barometer between turning points of the balance are used to evaluate momentary fluctuations in air pressure, which may have appreciable

effects on the turning points. The air temperature in the balance can be accurately measured since lag in thermometers is minimized by the extremely uniform temperatures. Relative humidity inside the balance case is indicated by an electric hygrometer. Once the values of these 1-kg standards are determined other standards of similar density can be compared with them without extensive measurements of air density.

In the high-precision calibration of sets of weights, the Bureau follows the general plan established by the International Bureau of Weights and Measures at Sèvres, France. In this method, a summation of the weights from a set is determined by comparison with a known standard, after which the weights are intercompared by a series of weighings consisting of all possible combinations of various loads. These weighings are adjusted by the method of least squares to give values for the larger weights of the set and for a summation of the smaller weights. This latter summation is then intercompared in similar fashion to obtain values for each of a group of smaller weights and for a smaller summation. In this way, values are eventually obtained for all the weights in the set, the last series of observations giving values for the smallest weights, which are used in determining the sensitivity of the balance.

In general, the values obtained for the smaller weights will be slightly different from the preliminary values that were used in sensitivity determinations for the various weighings. The new values for the sensitivity weights must therefore be substituted in the computations and the entire computations repeated. Usually after one recomputation the values obtained for the sensitivity weights are identical with those substituted for the sensitivity weights. Because this method accurately subdivides the value of the prototype kilogram to give the values of the sensitivity weights, the results are based entirely on the value of the national standard. This procedure is used in the calibration of the Bureau's best set of platinum-iridium standards from 500 grams to 1 milligram and for smaller aluminum weights down to 0.05 milligram. As standard weights certified by the Bureau are available in various denominations, it is not ordinarily necessary for other laboratories in this country to make these rather tedious computations.

The Bureau maintains accurate secondary standards for the avoirdupois pound, troy pound, and troy ounce, defined by their fixed ratios to the prototype kilogram. Groups of two 1-pound avoirdupois standards, or three 1-pound troy standards, or three 10-ounce troy standards are compared with the best secondary 1-kilogram standards, the differences in each case being made up with small standard weights from the best platinum-iridium metric set. From these standards of customary units, values are derived for standards used by the States in regulation of commerce and industry.

To provide standards for modern microbalances, the Bureau has recently developed methods for calibrating small standard weights below 100 milligrams with a precision of one or two ten-millionths of a gram. This precision—10 or 15 times as great as that now obtained



The national standard of mass, Prototype Kilogram No. 20, is a platinum-iridium cylinder 39 millimeters in diameter and 39 millimeters high. It is an accurate copy of the international standard kept at the International Bureau of Weights and Measures at Sèvres, France.

by indirect methods of calibration—will permit the weighing of samples about one-tenth as large as formerly. The resultant increase in the field of usefulness of microbalances should mean great savings to workers in such fields as atomic energy, where minute samples must be weighed with high accuracy.

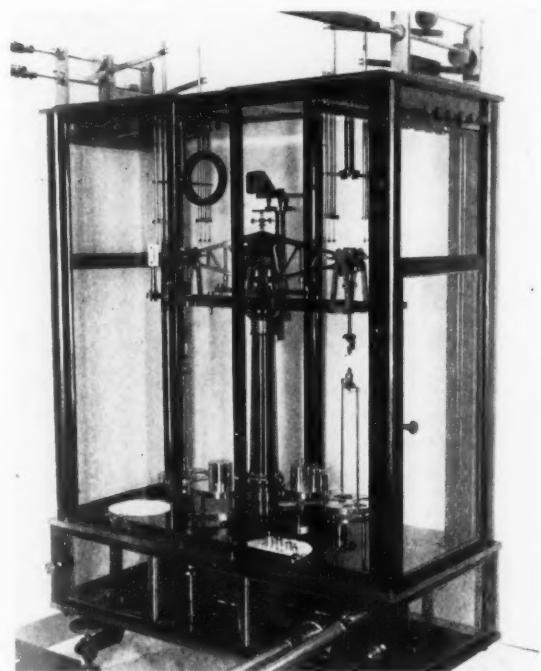
Calibration of these small weights is carried out on high-grade assay balances, using the method of double transposition, in which loads are interchanged twice and a small sensitivity weight, usually 0.1 or 0.05 milligram, is added to obtain a sensitivity deflection. Equilibrium points are computed from a series of five or more turning points observed through a reading telescope on a finely divided index scale. The sensitivity is such that 0.1 scale division is equal to about 0.2 microgram. Thus, a reading accuracy of about one-twentieth of a scale division or slightly better is readily obtained by experienced observers.

In order to certify values below 100 milligrams to an accuracy of 0.1 microgram, it is necessary to work down from 1-gram standards, intercomparing the weights to be calibrated in all possible combinations, and to repeat the entire computation to obtain values for the sensitivity weights, which are accurate subdivisions of the 1-gram standards. The reliability of the balances used is such that for weighings expertly carried out under optimum conditions the values obtained by least-square adjustment of the weighings are reproducible to 0.1 microgram. Moreover, discrepancies between observed and computed or adjusted values are usually not greater than 0.1 microgram. This accuracy, perhaps the highest ever obtained with the knife-edge type of balance, is of special value in the weighing of radioactive material, making it possible to handle

smaller samples with greater safety and to obtain small samples at less cost of time and labor.

Development of methods for calibration of extremely small weights has greatly simplified the problem of calibrating quartz microbalances directly in terms of known standard weights. This, in turn, makes possible the saving of much time, equipment, and labor formerly spent in indirect methods of calibration. The quartz microbalance itself is now being intensively studied as part of a broad program now being undertaken by the Bureau for the improvement of precision balances. Efforts are being made to improve the uniformity of its fabrication and to increase its capacity. The construction of the equal-arm knife-edge type of balance is also under study, with the object of attaining greater accuracy through improved design, use of new materials, and avoidance of troublesome temperature effects.

In recent years, the increasing interest in scientific research in this country has brought about a corresponding increase in the number of standard weights submitted to the Bureau for calibration. The largest part of this work involves sets of weights in the following combinations of units: 5, 3, 2, 1, 1; 5, 2, 2, 1, 1; and 5, 2, 1, 1, 1. At present all computations involved in the separate calibration schemes used for each of



The most accurate of the precision balances employed by the Bureau for calibration of secondary standards of mass is operated entirely by remote control to avoid errors due to the heat given off by the human body. By means of rods (above and lower right), the balance beam is arrested and released, the loads on the pans are interchanged, and sensitivity weights are added. A precision better than 1 part in 100,000,000 is obtained.

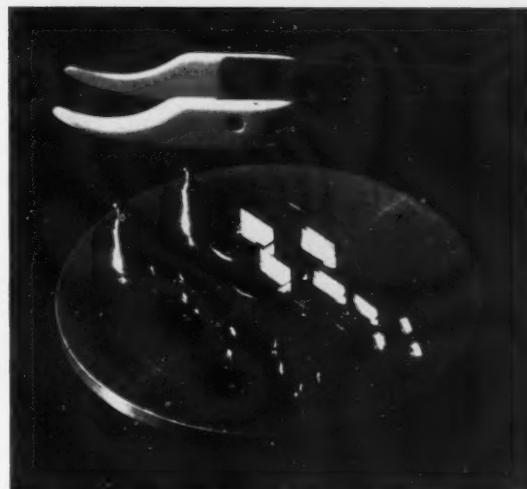
these combinations are performed with a slide rule or a desk calculator and are then checked in detail and reviewed. Plans are under way to do a large part of this work by means of a computing machine designed to give a simultaneous solution for the values of the weights from a series of equilibrium points as observed on a balance. For this purpose, a single series of observations has been devised by means of which weights in all three of the above combinations may be calibrated. Such a scheme makes possible the construction of a machine designed for the simultaneous solution of the one series of observations and its use to compute values for sets of weights in all three combinations of units. This should largely eliminate errors of computations and result in great savings of time and effort in the Bureau's weight-calibration program.

Commerce and Industry

As the custodian of the national standards of physical measurement, the National Bureau of Standards not only sets up and maintains the basic standards of length, mass, time, and other physical quantities, but also undertakes the necessary research leading to improvements and refinements in such standards and measurement methods. At the same time, the Bureau constructs, maintains, and intercompares reference and working standards that are calibrated in terms of the national standards and used in science, engineering, industry, and commerce. These services are performed for agencies of the Federal and State governments, scientific societies, educational institutions, and firms or individuals within the United States engaged in manufacturing or other pursuits requiring the use of standards or standard measuring instruments.

Weighing devices and weights employed by other Federal agencies, such as the weights used by the Bureau of Internal Revenue to weigh the products of distilleries or those used by the Bureau of Customs to weigh cargoes, are tested and calibrated directly by the National Bureau of Standards. Thus, through the introduction of the metric carat, based on the standard kilogram, uniformity in the measurement of the weight of gems has been achieved. Previously the many different values of the carat in use had caused much difficulty in customs work. Likewise, uniformity in the manufacture of drugs is now possible through the use of certified weights. Weights and balances tested by the National Bureau of Standards are used by the Food and Drug Administration in its regulatory work. In accordance with an Act of Congress dated March 4, 1911, the weight and fineness of coinage is verified each year through comparison with standard weights of the National Bureau of Standards.

Over 30 years ago the Bureau began the testing of railway track scales in order to provide a uniform basis for the weighing of carload shipments. At that time errors in track scales ranged from 30 to 21,600 pounds per 100,000, and it was not unusual for a load that was weighed at 100,000 pounds in one locality to be "weighed in" at its destination as 80,000 pounds. Discrepancies of this magnitude required check-weighing



Standard weights used by the Bureau for calibration of standards for modern microbalances. Values range from 1 gram to 0.05 milligram.

all along the route and, as the results of check-weighing were often not in agreement, caused much confusion. In the 34 years from 1914 to 1948, track scales meeting tolerance requirements increased from 33 to 84 percent of the total. During that time, a reduction in mean errors of the scales from 0.57 to 0.15 percent was effected.

Test weights certified by the Bureau for values of 500 pounds, 1,000 pounds, or more are used to determine the accuracy of large-capacity vehicle scales used in industry and commerce. With the rapidly expanding use of the highways and the increasing size of truck loads, scales of this kind have become increasingly important. As the result of a survey conducted by the Bureau in 1936, which showed that the vehicle scales of this country were not sufficiently accurate, the regulatory agencies throughout the United States have greatly improved the accuracy of their vehicle-scale testing methods. Large errors in transportation charges and much expensive litigation have thus been avoided.

Although the Constitution gave Congress the power "to fix the standard of weights and measures," weights used during the early days of the Republic were far from uniform. Not only was there much uncertainty as to the value of the pound, troy ounce, and other units, but the fineness of United States coinage had been questioned. To remedy the situation, a small Office of Standard Weights and Measures was set up in 1830 in the Treasury Department as part of the Coast Survey. Through this Office, definite values were established for the pound and other common units. Standard weights and balances and standards of length and capacity were constructed, and accurate copies were distributed to the various States and customs houses. In this way, a high degree of uniformity in standards was established and maintained. Finally, in 1901, in response to the growing need for a national scientific laboratory similar

to those of Germany and Great Britain, and in recognition of the high standards that had been established in weights and measures, the Congress enacted a law establishing the National Bureau of Standards.

The uniformity that the Bureau has been able to achieve in weights of all types and denominations—from 0.05-milligram weights for microbalances to 10,000-pound test weights for cargo scales—has been of great economic importance to the producing, manufacturing, processing, and distributing agencies of this country and to all purchasers of commodities. Essentially, the Congress has left to the individual States the regulation of commercial weighing devices and operations. In this work the National Bureau of Standards serves principally in an advisory capacity. However, uniformity between the States is maintained by the periodic comparison of State standards with the primary standards kept at the Bureau.

Office of Weights and Measures

To aid in the translation of the basic standards of mass to the channels of industry and commerce, the Bureau established in 1947 an Office of Weights and

Measures whose over-all function is to promote the extension, raise the standard of efficiency and coverage, and increase the degree of uniformity of State weights and measures supervision throughout the United States. A definite program of assistance to State and local departments of weights and measures, as well as to business and industry has been set up and successfully pursued.

A large part of the activity of the Office of Weights and Measures consists of consultative services rendered through correspondence; through visits to the Office by representatives of Federal agencies, business and manufacturing concerns, and weights and measures officials; and through visits of members of the Office to weights and measures officials in their own jurisdiction. The field of inquiries is broad, embracing the drafting of new legislation; the interpretation of laws, specifications, tolerances, and regulations; the design of testing equipment; methods of test of commercial equipment; the reporting of activities in different weights and measures jurisdictions; problems of and plans for weights and measures administration; planning and conducting weights and measures conferences; and training schools for State departments.

The McKee Worker-Consistometer With Constant-Speed Drives

Recent improvements in the McKee Worker-Consistometer, which was originally designed at the National Bureau of Standards to measure the effect of mechanical working on the physical properties of lubricating greases, permit operation at constant nominal rates of shear and extend the range to higher viscosities. With these modifications, the instrument has proved useful in studies of the rheological properties of rubber and rubber solutions, as well as greases and similar substances.

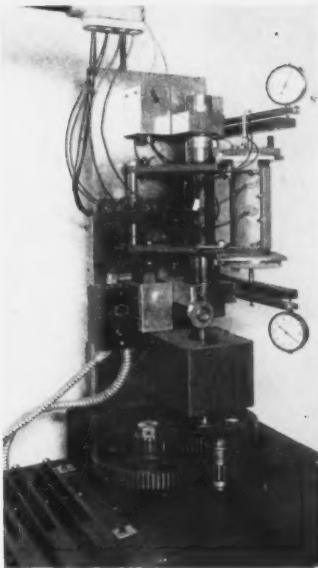
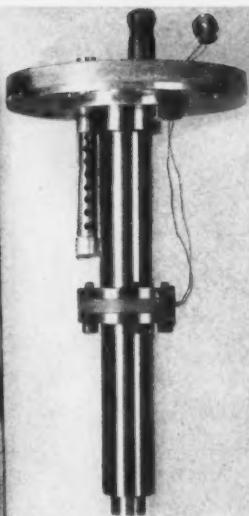
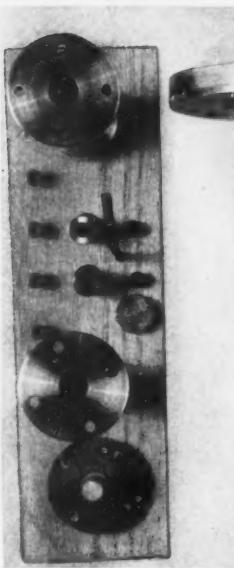
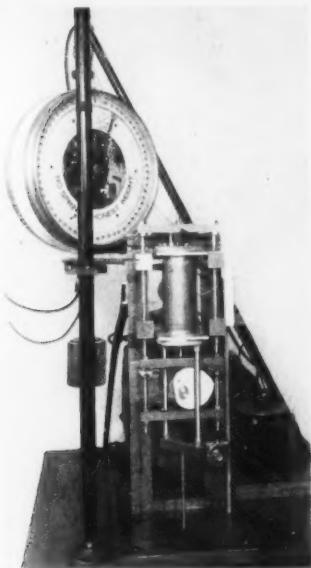
In its original form the worker-consistometer employs weights to provide various constant loads at which measurements are made of the rates of flow of the material under test. The modifications are chiefly in the driving mechanism and the method for measuring the forces involved. Two constant-speed drives have been constructed: one is a cam-operated mechanism with a load range up to 200 pounds, the other a screw-operated drive with a capacity of 1,600 pounds. The operation of each mechanism is automatic and is such that readings of the forces may be taken at intervals during the run of continual working.

The design of the major elements has been retained. The worker unit consists of two coaxial steel cylinders and mating pistons with a capillary-type shearing element between them. Relative motion between pistons and cylinders forces the test material through the shearing element. Each unit is provided with 1-hole, 10-hole, and 50-hole shearing elements, which are made by drilling holes in circular steel disks $\frac{1}{4}$ -inch thick,

using a No. 79 drill. Capillaries $\frac{1}{4}$ -inch long and about 0.015 inch in diameter are thus produced in the disks.

In the cam-operated apparatus, the pistons are moved back and forth, and determinations of the pressure developed in pushing the sample through the capillaries in the shearing element are made by measuring the force required to hold the cylinders in a fixed position. The worker unit is enclosed in a duralumin housing, which in turn is encircled by an electric heater. A mercury-in-glass thermal regulator controls the test temperature, which is measured by a thermocouple in contact with the shearing element. Flanges at the ends of the housing are attached to flexure plates, which provide "free" action along the vertical axis. Push rods, actuated by the cam through a connecting yoke, move the pistons back and forth a distance of approximately 2 inches. During the upward pass, the force required to push the material through the shearing element is measured by a weighing scale, which is connected to the cylinder assembly by rods acting through an 8-to-1 lever arm. The arrangement is such that the load range is from 1 to 200 pounds.

The driving mechanism and the series of disks provide a continuous range of nominal rates of shear from 200 to 100,000 reciprocal seconds. However, this range is limited for any given material by the load span of 1 to 200 pounds. For the entire range, this load span corresponds to viscosities of 1 to 100 poises; while for the relatively low rate of 200 reciprocal seconds, the viscosity range is from 20 to 4,000 poises.



Two constant-speed drives, constructed as modifications of the McKee workerconsistometer, permit operation at constant nominal rates of shear and extend the range to higher viscosities: (Left) A cam-operated mechanism with a load range up to 200 pounds; (right) a screw-operated drive with a capacity of 1,600 pounds; (center) major elements and assembled unit of the worker-consistometer.

In the screw-operated apparatus, the cylinders of the worker unit are moved back and forth, and the force measured is that required to hold the pistons in a fixed position. The unit is enclosed in a temperature-controlled steel housing, which is attached to a steel operating yoke so as to permit vertical motion but no rotation. This yoke is driven through a screw and nut by a reversible gear motor, and interchangeable gear trains provide changes in speed. Adjustable screws on the yoke contact push buttons, thus operating magnetic switches that automatically reverse the motor at the end of each upward and downward stroke of the yoke. The screws and push buttons are adjusted for a stroke of about 2 inches.

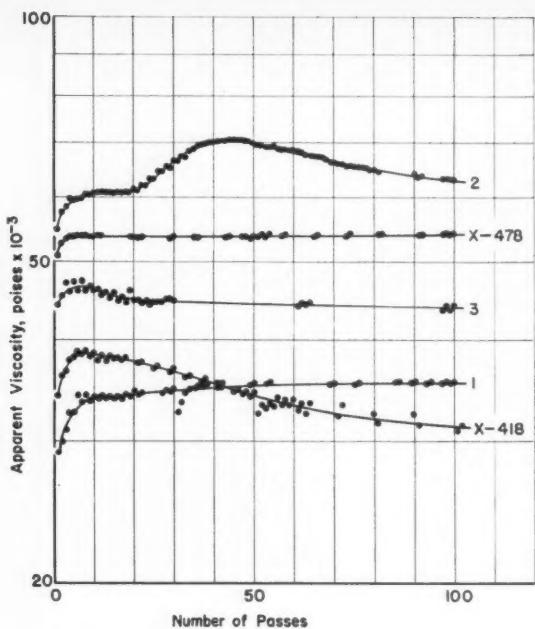
During the reciprocating motion of the cylinders, the pistons are held stationary by loading pins acting against calibrated steel springs. A dial indicator measures the deflection of the cantilever arm of the spring and hence the force necessary to push the test material through the capillaries in the shearing element. Five pairs of springs of different capacity cover the range of loads up to 1,600 pounds. The gear trains provide a number of rates of shear ranging from less than 1 up to 100,000 reciprocal seconds. The viscosity range is 3 times greater than that of the cam mechanism for its range of shear rate (200 to 100,000 reciprocal seconds). With the screw-operated apparatus, the range of viscosity determinations is extended to 600,000 poises at 10 reciprocal seconds or 6,000,000 poises at 1 reciprocal second.

In a typical test with the worker consistometer, for example, a lubricating grease operating at 100° F. was investigated at various constant rates of shear. Dur-

ing each run at a given rate of shear, flow measurements were taken at intervals throughout a period of mechanical working of 200 passes, after which the sample was transferred to a storage cylinder. After 4 weeks of storage at room temperature, flow measurements were again made with each sample during a working period of 26 passes. They were then subjected to an additional storage time of 35 weeks and another 26-pass working period.

With this grease, the effect of mechanical working in lowering the viscosity was quite marked during the first 200 passes. The change in viscosity was found to be roughly proportional at all rates of shear over the range covered. The data taken at the first pass after a storage period provide an indication of the so-called healing process. Generally, there is a measurable increase in viscosity after storage. Apparently the grease becomes relatively stable (with respect to mechanical working) after the first working period, since subsequent working after storage does not lower the viscosity much below that at the 200th pass.

With two other greases operating at a rate of shear of 13,100 reciprocal seconds and test temperatures of 100° and 245° F., apparent viscosities were determined at intervals during 226 passes with a four-week storage period after the 200th pass. With one grease the increase in temperature from 100° to 245° F. caused a very marked reduction in the viscosity of the grease in both the unworked and worked condition. On the other hand, when the second grease was worked at the higher temperature, its viscosity increased to such an extent that, at the 200th pass, there was not much difference in viscosity at the two test temperatures.



The McKee worker-consistometer has also provided important data on the effect of working on the viscosity of five GR-S rubbers: two in commercial production and three special experimental samples. In these tests,

The improved McKee worker-consistometer has proved useful for studying the effect of working on the apparent viscosity of five synthetic (GR-S) rubbers. Specimens 1, 2, and 3 are experimental samples; X-418 and X-478, commercial products. Temperature—212° F.; rate of shear—99 reciprocal seconds.

100 passes were made with each sample at a rate of shear of 99 reciprocal seconds and at a temperature of 212° F. There was an increase in viscosity in each case during the first few passes, after which the behavior was different for different samples.

One of the chief advantages of the worker-consistometer is that various materials may be worked and their flow characteristics measured in the same series of operations. The multi-hole disks give flexibility in covering wide ranges of rates of shear and consistency. Moreover, the constant-speed drives permit operation at constant rates of shear and extend the range to high viscosities. The investigations at the National Bureau of Standards indicate the potentialities of the apparatus in the study of the effect of temperature, rate of shear, and mechanical working upon the apparent viscosity of any non-Newtonian materials having thixotropic properties. In fact, the McKee worker-consistometer has already been used to determine consistencies of materials ranging from a light lubricating grease up to and including 100-percent raw rubber.

For further technical details, see The McKee worker-consistometer with constant-speed drives, by S. A. McKee and Hobart S. White, *J. Research NBS*, **46**, 18 (1951) RP2710. The earlier model of the worker-consistometer is described in *NBS Technical News Bulletin* **33**, 10 (1949); and *ASTM Bulletin* No. 153, p. 90 (August 1948).

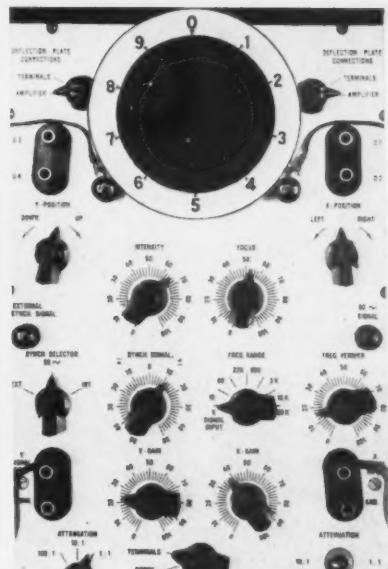
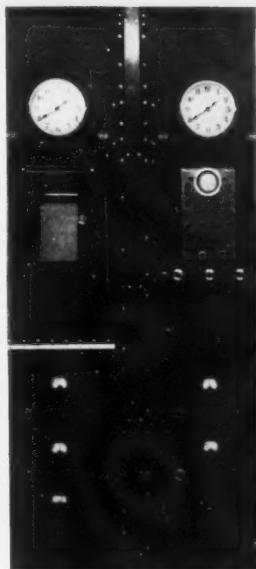
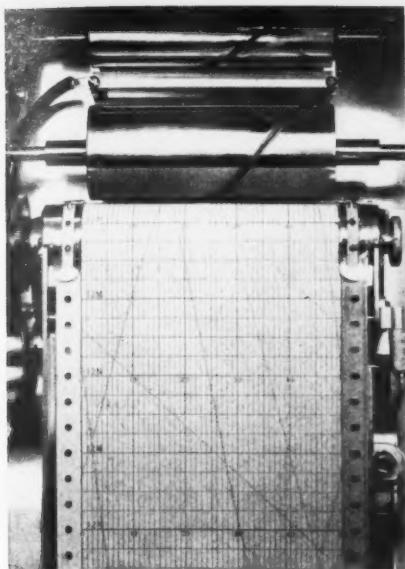
New Spark Chronograph and Chronoscope

The National Bureau of Standards' system for monitoring the precision time-keeping of a group of standard crystal clocks has been further refined by J. M. Shaull and C. M. Kortman of the Bureau staff. This refinement consists of an improved spark chronograph and chronoscope, which together record time differences as small as 20 millionths of a second. The new development may be easily applied to checking stability of oscillators and frequency dividers and rating clocks and chronometers over long periods of time. The method is now being applied, in principle, by watchmakers for the rapid adjustment of watches and clocks.

The spark chronograph records time differences of two clocks to 1 millisecond by linearly sweeping a spark discharge point across the waxed paper strip of a specially designed recorder. The chronoscope uses the visual characteristics of the cathode-ray tube to increase the resolution of the chronograph to 0.02 millisecond. Together these instruments constitute a reference clock with which all other crystal clocks comprising the primary standard, including those of the NBS radio station WWV, may be intercompared.

The improved spark chronograph includes a single-turn helix wound on a rotating drum driven by a synchronous motor. Beneath the drum is an insulated knife edge over which a strip of waxed paper slowly

passes. A high-voltage pulse causes a spark to jump from the knife edge to the nearest point of the helix, perforating the paper, melting the wax and thus leaving a permanent record. Should the motor driving the drum be supplied by subfrequencies from a standard oscillator while the high-voltage pulse is controlled by another crystal clock, a recorded picture of the relative rates is readily obtained. If the drum-control frequency is equal to the spark-control frequency, each time a spark occurs the rotating drum turns through an angle which is an exact multiple of 360°. Thus, the same point on the helix will be opposite the knife edge, and the record on the waxed paper will be straight line running vertically up the chart. However, should there be a difference in frequencies, the drum will rotate through a greater or smaller angle, causing a different point on the helix to be nearest the knife edge at the time of the spark. As a result, the record will slope to the right if the clock controlling the spark is running faster, or to the left if the clock is running slower. The difference in rates may be evaluated by measuring the amount of displacement over a given period. If the spark-generating equipment is switched, in turn, to each of several clocks, the chronograph provides a convenient method of intercomparing and recording their operation. In practice, a motor-driven switching unit



The improved spark chronograph and chronoscope are shown in the rack (center). The spark chronograph (left) consists essentially of a one-second and 1/10-second drum (top of case). A spark from a knife edge behind the drum strikes the helix and pierces the waxed paper recording strip. The chronoscope (right) visually indicates the differences between two clocks to within 0.02 millisecond.

connects each clock to the spark generator every 15 minutes. In addition, push buttons permit manual checking of a particular clock at any time.

The rotating drum is made of stainless steel; a helical groove is cut into its surface, and a steel spline is soldered into this groove. The synchronous motor drives the drum at a rate of 10 revolutions per second, causing the helix to cover a time interval of 0.1 second per sweep. The length of the drum is 5 inches, and the paper is of the same width; thus 0.001 second is represented by 0.05 inch across the paper chart. The chart is ruled with 10 lines per inch, and values are easily interpolated to the nearest millisecond.

Because the drum rotates at 10 revolutions per second, some method is needed to indicate in which tenth of a second the helix is turning. For this purpose, a smaller drum is mounted above the major one and is geared down to rotate at a speed one-tenth as great, and passes over another knife edge that is divided into 10 parts. A switch is provided so that the high-voltage pulses can be applied to the smaller drum and the tenths position noted visually.

A pulse-shaping circuit provides the desired pulse for spark generation with different types of input signals. Tests with sine-wave input have shown that the circuit will operate between 10 and 400 cycles when less than 10 volts rms is applied, and over a much wider range with greater amplitude.

As the chronograph records only to 0.5 millisecond, the chronoscope was developed primarily as a vernier device to permit the determination of long intervals with greater accuracy. In this instrument, the output of one crystal clock and frequency divider is used to produce a circular sweep with small, fixed marker dots

on the face of a 3-inch cathode-ray tube. A pulse from another crystal clock produces a large bright spot on the sweep. By observing the position of this spot in relation to the marker dots, it is possible to measure relative time changes within a very small fraction of the interval required for one circular sweep. The time base and marker frequencies are obtained from the same frequency dividers that supply the chronograph drive, so that the chronograph and chronoscope are locked in time phase just as are the minute and sweep-second hands of a conventional clock mechanism.

The circular sweep is obtained by applying 100-cycle voltages in phase quadrature to the deflection plates of the cathode-ray tube. Thus a 360° sweep is accomplished in 0.01 second. The grid of the cathode-ray tube is biased below cut-off, so that no trace appears on the face of the tube except when a positive pulse is applied with sufficient amplitude to let the tube conduct. A 10-kilocycle signal applied to the grid therefore produces a circle composed of 100 dots, each 0.1 millisecond apart. Reference points are produced by a 1-kilocycle sine wave shaped to give negative pulses just wide enough to blank out every tenth one of the 10-kilocycle dots. Thus the cathode-ray tube shows ten groups of nine dots, each separated by a blank space. The signal to be measured is fed to the chronoscope as a strong positive pulse, which places an enlarged dot on the face of the tube. The entire circular sweep represents 0.01 second, while the distance between dots corresponds to an interval of 0.1 millisecond. By estimating fifths between the small dots, the position of the larger dot may be determined with sufficient accuracy to measure the change in relative time, as kept by two clocks, within 0.02 millisecond.

For greater resolution a larger cathode-ray tube may be used to increase the span between adjacent markers. For greater accuracy, 100-kilocycle markers could also be added. Another possibility is the use of a second chronoscope starting with either a 1-kilocycle or 10-kilocycle time base and using 100-kilocycle or 1-megacycle markers to permit higher resolution.

Precise measurements of time and frequency are becoming increasingly important in many technical fields—for example, in long-range radio navigation systems, in the upper range of the microwave region where atomic systems can serve as electronic components, and in basic research in microwave spectroscopy and molecular structure.

Publications of the National Bureau of Standards

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CRPL-D76. Basic Radio Propagation Predictions for March 1951. Three months in advance. Issued December 1950. 10 cents.

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